MEASUREMENT OF HIGH PURITY METALS USING THE NU ASTRUM GD-MS: ABUNDANCE SENSITIVITY

INTRODUCTION

Glow discharge mass spectrometry (GD-MS) is a powerful analytical technique designed for the quantitative determination of trace level impurities in solid matrices. These are often ultra-pure materials for the semiconductor industry. GD-MS instruments must be capable of detecting the trace impurities to very low (sub mg/Kg) levels. At these levels any non-matrix species detected may originate either from the sample being measured or else from molecular species created in the instrument source region. It is common for trace impurities and molecular species to have approximately the same mass, meaning identification can be difficult. This can restrict the analytical capability of the instrument and is the reason why high resolution is often employed for GD-MS instruments.

An additional problem to overcome is that a small analyte peak can easily be hidden beneath the "tail" of a much larger peak. The tailing is defined as abundance sensitivity, meaning the fraction of an analyte peak that remains at one mass unit separation from that peak. Improving the abundance sensitivity of a GD-MS instrument leads to reduced tailing of peaks and ensures better baseline separation and therefore better detection limits. There are various design characteristics to be considered to ensure a good abundance sensitivity. These are often related to the vacuum pumping system, the vacuum housing construction and the ion optical path design. However, a full discussion is beyond the scope of this application note. Here we report the abundance sensitivity of the Nu Instruments Astrum GD-MS.

Instrumentation

The Nu Astrum is the latest generation of GD-MS instruments developed to be the benchmark in GD-MS. The instrument was designed in conjunction with the users of the most widely-used GD-MS, the VG9000. The best design concepts of the VG9000 were combined with advances in sample cell design, control electronics and pumping technology to produce a high performance new instrument designed specifically for ultra-trace analysis of impurities. Every effort has been made to ensure that the background level in the instrument is as low as possible, including cryo-cooling of the source.

Experiment

To measure the abundance sensitivity of the Nu Astrum GD-MS a high purity Cu pin sample was used. With the instrument working under normal operating conditions, the ion beam signal was measured at the ^{63}Cu mass position. The same measurement was taken at exactly one mass unit less to establish the degree of tailing. Note that the ^{63}Cu peak was sufficient to saturate the electron multiplier and the ion beam was therefore measured on the Faraday detector, whereas the scan at one mass unit less was measured using the electron multiplier. The electron multiplier efficiency was measured at 80%, and the instrument was operated at 4,000 resolution.

Discussion

To determine the abundance sensitivity we measured the ion beam intensity at the ^{63}Cu mass position (figure 1) and then measured the ion beam intensity at one mass unit lower (figure 2). The inverse ratio of the two values defines the abundance sensitivity. The relative efficiency of the electron multiplier as compared to the Faraday was taken in to account in the calculation. In this case a value of 160ppb was obtained.

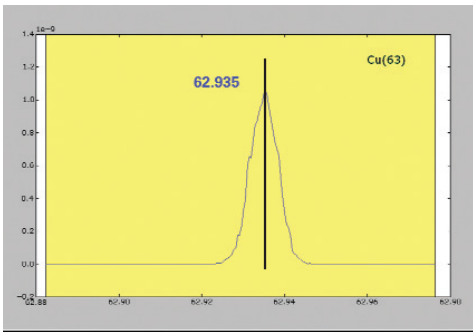

Figure 1: *spectra showing intensity of the ^{63}Cu peak*

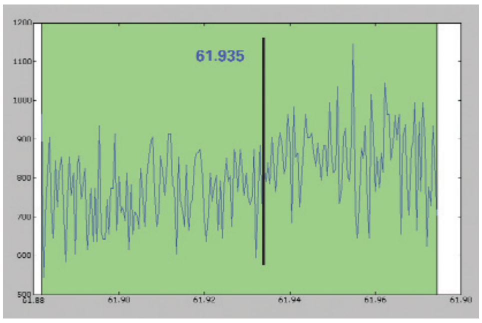

Figure 2: *spectra at one mass unit from the ^{63}Cu peak*

Conclusions

The Nu Astrum is a new high performance GD-MS that has been tailored to the exacting requirements of the high purity materials industry. The instrument has extremely low detection limits making it suitable for the analysis of even the most refined metals. We have shown that the abundance sensitivity for copper is ~160ppb.